

N-Benzoyl-N',N''-dicyclohexyl-phosphoric triamide

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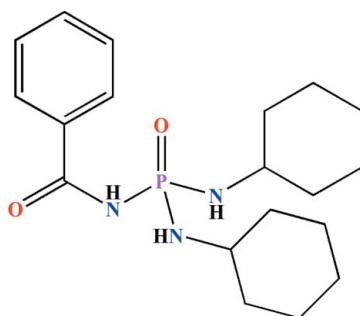
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Key indicators: single-crystal X-ray study; $T = 293\text{ K}$; mean $\sigma(\text{C}-\text{C}) = 0.008\text{ \AA}$; R factor = 0.051; wR factor = 0.122; data-to-parameter ratio = 11.5.

In the title compound, $\text{C}_{19}\text{H}_{30}\text{N}_3\text{O}_2\text{P}$, the central P atom has a distorted tetrahedral configuration. The N atoms in both cyclohexylamide moieties exhibit a slight deviation [0.32 (7) and 0.44 (6) \AA] from planarity, while the benzoylamide N atom is planar [0.11 (3) \AA]. In the crystal, molecules are linked via $\text{N}-\text{H}\cdots\text{O}(\text{P})$ and $\text{N}-\text{H}\cdots\text{O}(\text{C})$ hydrogen bonds, forming $R_2^2(10)$ rings within linear arrangements parallel to the b axis.

Related literature

For the synthesis and a spectroscopic study of title compound, see: Gholivand *et al.* (2006). For bond lengths in related structures, see: Sabbaghi *et al.* (2010); Rudd *et al.* (1996). For hydrogen-bond motifs, see: Etter *et al.* (1990); Bernstein *et al.* (1995).



Experimental

Crystal data

$\text{C}_{19}\text{H}_{30}\text{N}_3\text{O}_2\text{P}$
 $M_r = 363.43$
Monoclinic, Cc
 $a = 20.9904 (17)\text{ \AA}$
 $b = 5.1503 (2)\text{ \AA}$
 $c = 21.1125 (18)\text{ \AA}$
 $\beta = 121.955 (11)^\circ$

$V = 1936.5 (2)\text{ \AA}^3$
 $Z = 4$
Cu $K\alpha$ radiation
 $\mu = 1.39\text{ mm}^{-1}$
 $T = 293\text{ K}$
 $0.28 \times 0.05 \times 0.01\text{ mm}$

Data collection

Oxford Diffraction Xcalibur Ruby Gemini diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
 $T_{\min} = 0.978$, $T_{\max} = 1.000$

2758 measured reflections
2758 independent reflections
2294 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.064$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.051$
 $wR(F^2) = 0.122$
 $S = 1.05$
2758 reflections
239 parameters
2 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.46\text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.18\text{ e \AA}^{-3}$
Absolute structure: Flack (1983), 908 Friedel pairs
Flack parameter: 0.11 (4)

Table 1
Hydrogen-bond geometry (\AA , $^\circ$).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
N4—H4 \cdots O3 ⁱ	0.90 (4)	2.16 (4)	2.988 (4)	154 (3)
N5—H5 \cdots O2 ⁱⁱ	0.77 (5)	2.30 (6)	3.019 (5)	156 (6)

Symmetry codes: (i) $x, y + 1, z$; (ii) $x, y - 1, z$.

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SUPERFLIP* (Palatinus & Chapuis, 2007); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LD2010).

References

- Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
- Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst. B* **46**, 256–262.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Flack, H. D. (1983). *Acta Cryst. A* **39**, 876–881.
- Gholivand, K., Mojahed, F., Madani Alizadehgan, A. & Bijanzadeh, H. R. (2006). *Z. Anorg. Allg. Chem.* **632**, 1570–1577.
- Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
- Oxford Diffraction (2010). *CrysAlis PRO*. Oxford Diffraction Ltd, Yarnton, England.
- Palatinus, L. & Chapuis, G. (2007). *J. Appl. Cryst.* **40**, 786–790.
- Rudd, M. D., Lindeman, S. V. & Husebye, S. (1996). *Acta Chem. Scand.* **50**, 759–774.
- Sabbaghi, F., Pourayoubi, M., Toghraee, M. & Divjakovic, V. (2010). *Acta Cryst. E* **66**, o344.
- Sheldrick, G. M. (2008). *Acta Cryst. A* **64**, 112–122.

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N-Benzoyl-N',N'-dicyclohexylphosphoric triamide

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Comment

The synthesis of the title compound, $C_6H_5C(O)NHP(O)[NHC_6H_{11}]_2$, was previously published by Gholivand *et al.* (2006). Here, we report its crystal structure (Fig. 1).

The P=O, C=O and P—N bond lengths match those found for other compounds with the [(N)(N)P(O)NHC(O)] skeleton (Sabbaghi *et al.*, 2010). The nitrogen atoms show sp^2 character and the environment of N atom in C(O)NHP(O) moiety is practically planar. The tetrahedral configuration of phosphorus atom is significantly distorted (Fig. 1) as it has been noted for other phosphoric triamides and their chalco-derivatives (Rudd *et al.*, 1996): the bond angles at the P atom vary in the range from 102.7 (2) $^\circ$ to 117.2 (2) $^\circ$, while the P—N bond distances range from 1.615 (4) to 1.730 (4) Å. Cyclohexyl groups are in a chair conformation with the adjacent NH groups oriented equatorially.

The NH unit of the C(O)NHP(O) moiety adopts a *syn* orientation with respect to the phosphoryl group; moreover, the NH units of two $C_6H_{11}NH$ moieties are in a *syn* orientation with respect to each other.

In the crystal structure, the molecules are linked *via* N—H \cdots O=P and N—H \cdots O=C hydrogen bonds, in which carbonyl oxygen interacts with benzamide N—H and P(O) group binds to a cyclohexylamido moiety. This way, $R_2^2(10)$ rings are built (Etter *et al.*, 1990; Bernstein *et al.*, 1995), that are further connected in linear arrangements along y axis (Table 1, Fig. 2).

Experimental

$C_6H_5C(O)NHP(O)[NHC_6H_{11}]_2$ was prepared according to the procedure reported by Gholivand *et al.* (2006). Single crystals of title compound were obtained from CH₃OH after slow evaporation at room temperature.

Refinement

At the end of the refinement the highest peak in the electron density was 0.460 e Å $^{-3}$, while the deepest hole was -0.180 e Å $^{-3}$.

Figures

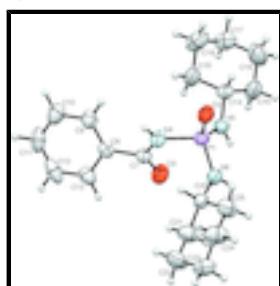


Fig. 1. An ORTEP-style plot of title compound with labeling. Ellipsoids are given at the 50% probability level.

supplementary materials

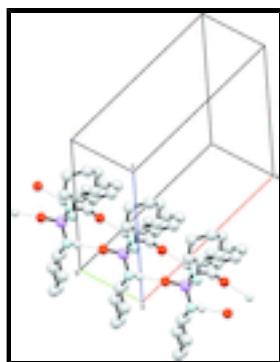


Fig. 2. Partial packing view showing the formation of the chain through N—H···O hydrogen bonds (along the b axis) which are shown as dotted lines. H atoms not involved in the hydrogen bondings are omitted.

N-Benzoyl-N',N''-dicyclohexylphosphoric triamide

Crystal data

C ₁₉ H ₃₀ N ₃ O ₂ P	$F(000) = 784$
$M_r = 363.43$	$D_x = 1.247 \text{ Mg m}^{-3}$
Monoclinic, Cc	Cu $K\alpha$ radiation, $\lambda = 1.54180 \text{ \AA}$
Hall symbol: C -2yc	Cell parameters from 1733 reflections
$a = 20.9904 (17) \text{ \AA}$	$\theta = 4.2\text{--}70.2^\circ$
$b = 5.1503 (2) \text{ \AA}$	$\mu = 1.39 \text{ mm}^{-1}$
$c = 21.1125 (18) \text{ \AA}$	$T = 293 \text{ K}$
$\beta = 121.955 (11)^\circ$	Prismatic, colorless
$V = 1936.5 (2) \text{ \AA}^3$	$0.28 \times 0.05 \times 0.01 \text{ mm}$
$Z = 4$	

Data collection

Oxford Diffraction Xcalibur Ruby Gemini diffractometer	2758 independent reflections
Radiation source: Enhance (Cu) X-ray Source graphite	2294 reflections with $I > 2\sigma(I)$
Detector resolution: 10.2673 pixels mm^{-1}	$R_{\text{int}} = 0.064$
ω scans	$\theta_{\text{max}} = 70.3^\circ$, $\theta_{\text{min}} = 4.8^\circ$
Absorption correction: multi-scan (<i>CrysAlis PRO</i> ; Oxford Diffraction, 2010)	$h = -24 \rightarrow 24$
$T_{\text{min}} = 0.978$, $T_{\text{max}} = 1.000$	$k = 0 \rightarrow 6$
2758 measured reflections	$l = -25 \rightarrow 25$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.051$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.122$	$w = 1/[\sigma^2(F_{\text{o}}^2) + (0.0489P)^2]$

$S = 1.05$	where $P = (F_o^2 + 2F_c^2)/3$
2758 reflections	$(\Delta/\sigma)_{\max} < 0.001$
239 parameters	$\Delta\rho_{\max} = 0.46 \text{ e } \text{\AA}^{-3}$
2 restraints	$\Delta\rho_{\min} = -0.18 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 908 Friedel pairs Flack parameter: 0.11 (4)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
P1	0.00072 (6)	0.2629 (2)	0.30701 (6)	0.0403 (2)
O2	-0.02706 (18)	0.5292 (5)	0.30029 (19)	0.0504 (8)
O3	0.11281 (18)	-0.1251 (6)	0.3206 (2)	0.0578 (9)
N4	0.0865 (2)	0.3029 (6)	0.3150 (2)	0.0435 (8)
N5	-0.0520 (2)	0.0724 (7)	0.2376 (2)	0.0438 (9)
N6	0.0150 (2)	0.0959 (8)	0.3781 (2)	0.0458 (9)
C7	0.1289 (2)	0.1008 (8)	0.3169 (2)	0.0398 (9)
C8	0.1958 (2)	0.1615 (7)	0.3117 (2)	0.0401 (9)
C9	0.1990 (3)	0.3714 (9)	0.2727 (3)	0.0490 (11)
H9	0.1598	0.4905	0.2518	0.059*
C10	0.2585 (3)	0.4083 (11)	0.2640 (3)	0.0616 (13)
H10	0.2590	0.5492	0.2367	0.074*
C11	0.3180 (3)	0.2347 (12)	0.2961 (3)	0.0677 (14)
H11	0.3587	0.2600	0.2907	0.081*
C12	0.3169 (3)	0.0239 (12)	0.3362 (3)	0.0722 (16)
H12	0.3569	-0.0923	0.3577	0.087*
C13	0.2559 (3)	-0.0130 (10)	0.3440 (3)	0.0533 (12)
H13	0.2549	-0.1546	0.3708	0.064*
C14	-0.0945 (2)	0.1484 (9)	0.1595 (2)	0.0451 (10)
H14	-0.1203	0.3120	0.1554	0.054*
C15	-0.1535 (3)	-0.0560 (13)	0.1154 (3)	0.0727 (17)
H15A	-0.1872	-0.0665	0.1336	0.087*
H15B	-0.1292	-0.2234	0.1235	0.087*
C16	-0.1994 (3)	0.0014 (16)	0.0314 (3)	0.0846 (19)
H16A	-0.2337	-0.1413	0.0055	0.102*

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H16B	-0.2290	0.1571	0.0225	0.102*
C17	-0.1502 (3)	0.0381 (11)	0.0011 (3)	0.0638 (13)
H17B	-0.1806	0.0862	-0.0511	0.077*
H17A	-0.1251	-0.1241	0.0045	0.077*
C18	-0.0922 (4)	0.2463 (12)	0.0436 (4)	0.0774 (17)
H18A	-0.1172	0.4125	0.0352	0.093*
H18B	-0.0589	0.2579	0.0250	0.093*
C19	-0.0464 (3)	0.1896 (13)	0.1269 (3)	0.0683 (16)
H19A	-0.0162	0.0356	0.1356	0.082*
H19B	-0.0124	0.3335	0.1525	0.082*
C20	0.0677 (2)	0.1637 (8)	0.4557 (2)	0.0426 (9)
H20	0.0962	0.3151	0.4562	0.051*
C21	0.1229 (3)	-0.0486 (12)	0.4978 (3)	0.0685 (15)
H21A	0.1533	-0.0758	0.4763	0.082*
H21B	0.0956	-0.2081	0.4916	0.082*
C22	0.1746 (3)	0.0067 (15)	0.5810 (3)	0.0805 (19)
H22A	0.2054	-0.1448	0.6055	0.097*
H22B	0.2077	0.1498	0.5878	0.097*
C23	0.1303 (3)	0.0742 (10)	0.6167 (3)	0.0650 (14)
H23B	0.1019	-0.0762	0.6155	0.078*
H23A	0.1646	0.1218	0.6685	0.078*
C24	0.0777 (4)	0.2951 (13)	0.5762 (3)	0.0771 (18)
H24A	0.1068	0.4505	0.5829	0.093*
H24B	0.0477	0.3271	0.5979	0.093*
C25	0.0261 (3)	0.2433 (12)	0.4937 (3)	0.0654 (15)
H25A	-0.0029	0.3987	0.4697	0.078*
H25B	-0.0088	0.1066	0.4868	0.078*
H4	0.1046 (19)	0.457 (7)	0.3124 (19)	0.015 (8)*
H6	0.014 (3)	-0.052 (9)	0.371 (3)	0.037 (13)*
H5	-0.038 (3)	-0.067 (11)	0.248 (3)	0.062 (17)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0447 (5)	0.0336 (4)	0.0472 (5)	0.0016 (5)	0.0276 (4)	0.0017 (5)
O2	0.0546 (17)	0.0351 (15)	0.071 (2)	0.0019 (13)	0.0401 (17)	0.0037 (14)
O3	0.061 (2)	0.0334 (17)	0.089 (3)	0.0009 (14)	0.047 (2)	0.0017 (15)
N4	0.0453 (19)	0.0326 (18)	0.060 (2)	0.0006 (15)	0.0333 (18)	-0.0008 (15)
N5	0.048 (2)	0.035 (2)	0.047 (2)	0.0003 (16)	0.0244 (18)	0.0047 (16)
N6	0.054 (2)	0.044 (2)	0.046 (2)	-0.0048 (18)	0.0301 (19)	-0.0002 (17)
C7	0.0389 (19)	0.036 (2)	0.045 (2)	0.0004 (17)	0.0228 (18)	0.0000 (17)
C8	0.041 (2)	0.0367 (19)	0.042 (2)	0.0018 (17)	0.0213 (18)	-0.0041 (17)
C9	0.052 (3)	0.045 (2)	0.059 (3)	0.002 (2)	0.035 (2)	-0.003 (2)
C10	0.068 (3)	0.062 (3)	0.076 (3)	-0.003 (3)	0.053 (3)	-0.003 (3)
C11	0.055 (3)	0.074 (4)	0.091 (4)	-0.011 (3)	0.050 (3)	-0.019 (3)
C12	0.037 (3)	0.076 (4)	0.091 (4)	0.011 (2)	0.026 (3)	-0.016 (3)
C13	0.046 (2)	0.049 (3)	0.057 (3)	0.006 (2)	0.022 (2)	-0.002 (2)
C14	0.048 (2)	0.042 (2)	0.045 (3)	0.0027 (19)	0.024 (2)	0.0002 (19)

C15	0.064 (3)	0.103 (5)	0.050 (3)	-0.034 (3)	0.030 (3)	-0.006 (3)
C16	0.059 (3)	0.116 (5)	0.064 (4)	-0.015 (3)	0.023 (3)	-0.008 (4)
C17	0.079 (3)	0.057 (3)	0.052 (3)	0.003 (3)	0.032 (3)	0.004 (2)
C18	0.107 (4)	0.070 (4)	0.067 (4)	-0.019 (4)	0.054 (4)	-0.003 (3)
C19	0.060 (3)	0.091 (4)	0.056 (3)	-0.024 (3)	0.033 (3)	0.001 (3)
C20	0.045 (2)	0.041 (2)	0.045 (2)	0.0004 (18)	0.025 (2)	0.0032 (18)
C21	0.074 (3)	0.077 (4)	0.057 (3)	0.027 (3)	0.037 (3)	0.010 (3)
C22	0.065 (3)	0.114 (5)	0.051 (4)	0.028 (4)	0.022 (3)	0.018 (3)
C23	0.083 (4)	0.055 (3)	0.053 (3)	-0.001 (3)	0.034 (3)	0.004 (2)
C24	0.102 (5)	0.075 (4)	0.056 (3)	0.017 (4)	0.043 (3)	0.000 (3)
C25	0.071 (3)	0.078 (4)	0.057 (3)	0.023 (3)	0.040 (3)	0.006 (3)

Geometric parameters (\AA , $^\circ$)

P1—O2	1.468 (3)	C16—C17	1.487 (8)
P1—N6	1.615 (4)	C16—H16A	0.9700
P1—N5	1.618 (4)	C16—H16B	0.9700
P1—N4	1.730 (4)	C17—C18	1.509 (8)
O3—C7	1.225 (5)	C17—H17B	0.9700
N4—C7	1.356 (5)	C17—H17A	0.9700
N4—H4	0.90 (4)	C18—C19	1.521 (9)
N5—C14	1.454 (6)	C18—H18A	0.9700
N5—H5	0.77 (5)	C18—H18B	0.9700
N6—C20	1.453 (6)	C19—H19A	0.9700
N6—H6	0.78 (4)	C19—H19B	0.9700
C7—C8	1.499 (6)	C20—C21	1.496 (7)
C8—C9	1.382 (6)	C20—C25	1.521 (6)
C8—C13	1.397 (6)	C20—H20	0.9800
C9—C10	1.366 (7)	C21—C22	1.525 (8)
C9—H9	0.9300	C21—H21A	0.9700
C10—C11	1.387 (8)	C21—H21B	0.9700
C10—H10	0.9300	C22—C23	1.514 (8)
C11—C12	1.383 (9)	C22—H22A	0.9700
C11—H11	0.9300	C22—H22B	0.9700
C12—C13	1.389 (7)	C23—C24	1.498 (8)
C12—H12	0.9300	C23—H23B	0.9700
C13—H13	0.9300	C23—H23A	0.9700
C14—C19	1.507 (7)	C24—C25	1.511 (8)
C14—C15	1.513 (7)	C24—H24A	0.9700
C14—H14	0.9800	C24—H24B	0.9700
C15—C16	1.533 (8)	C25—H25A	0.9700
C15—H15A	0.9700	C25—H25B	0.9700
C15—H15B	0.9700		
O2—P1—N6	117.2 (2)	C16—C17—C18	111.1 (5)
O2—P1—N5	115.7 (2)	C16—C17—H17B	109.4
N6—P1—N5	102.73 (19)	C18—C17—H17B	109.4
O2—P1—N4	103.77 (18)	C16—C17—H17A	109.4
N6—P1—N4	107.5 (2)	C18—C17—H17A	109.4
N5—P1—N4	109.79 (19)	H17B—C17—H17A	108.0

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C7—N4—P1	123.0 (3)	C17—C18—C19	111.6 (5)
C7—N4—H4	113 (2)	C17—C18—H18A	109.3
P1—N4—H4	124 (2)	C19—C18—H18A	109.3
C14—N5—P1	125.4 (3)	C17—C18—H18B	109.3
C14—N5—H5	120 (4)	C19—C18—H18B	109.3
P1—N5—H5	109 (4)	H18A—C18—H18B	108.0
C20—N6—P1	125.2 (3)	C14—C19—C18	112.8 (5)
C20—N6—H6	113 (4)	C14—C19—H19A	109.0
P1—N6—H6	112 (4)	C18—C19—H19A	109.0
O3—C7—N4	122.2 (4)	C14—C19—H19B	109.0
O3—C7—C8	120.2 (4)	C18—C19—H19B	109.0
N4—C7—C8	117.6 (3)	H19A—C19—H19B	107.8
C9—C8—C13	118.6 (4)	N6—C20—C21	112.4 (4)
C9—C8—C7	123.4 (4)	N6—C20—C25	110.6 (4)
C13—C8—C7	117.9 (4)	C21—C20—C25	111.4 (4)
C10—C9—C8	121.6 (5)	N6—C20—H20	107.4
C10—C9—H9	119.2	C21—C20—H20	107.4
C8—C9—H9	119.2	C25—C20—H20	107.4
C9—C10—C11	119.7 (5)	C20—C21—C22	113.7 (5)
C9—C10—H10	120.1	C20—C21—H21A	108.8
C11—C10—H10	120.1	C22—C21—H21A	108.8
C12—C11—C10	120.2 (5)	C20—C21—H21B	108.8
C12—C11—H11	119.9	C22—C21—H21B	108.8
C10—C11—H11	119.9	H21A—C21—H21B	107.7
C11—C12—C13	119.6 (5)	C23—C22—C21	111.5 (5)
C11—C12—H12	120.2	C23—C22—H22A	109.3
C13—C12—H12	120.2	C21—C22—H22A	109.3
C12—C13—C8	120.3 (5)	C23—C22—H22B	109.3
C12—C13—H13	119.9	C21—C22—H22B	109.3
C8—C13—H13	119.9	H22A—C22—H22B	108.0
N5—C14—C19	113.5 (4)	C24—C23—C22	110.6 (5)
N5—C14—C15	108.8 (4)	C24—C23—H23B	109.5
C19—C14—C15	110.2 (4)	C22—C23—H23B	109.5
N5—C14—H14	108.1	C24—C23—H23A	109.5
C19—C14—H14	108.1	C22—C23—H23A	109.5
C15—C14—H14	108.1	H23B—C23—H23A	108.1
C14—C15—C16	112.7 (5)	C23—C24—C25	112.7 (5)
C14—C15—H15A	109.1	C23—C24—H24A	109.1
C16—C15—H15A	109.1	C25—C24—H24A	109.1
C14—C15—H15B	109.1	C23—C24—H24B	109.1
C16—C15—H15B	109.1	C25—C24—H24B	109.1
H15A—C15—H15B	107.8	H24A—C24—H24B	107.8
C17—C16—C15	111.6 (5)	C24—C25—C20	113.2 (5)
C17—C16—H16A	109.3	C24—C25—H25A	108.9
C15—C16—H16A	109.3	C20—C25—H25A	108.9
C17—C16—H16B	109.3	C24—C25—H25B	108.9
C15—C16—H16B	109.3	C20—C25—H25B	108.9
H16A—C16—H16B	108.0	H25A—C25—H25B	107.8
O2—P1—N4—C7	-174.9 (4)	C7—C8—C13—C12	175.4 (4)

N6—P1—N4—C7	60.3 (4)	P1—N5—C14—C19	74.0 (5)
N5—P1—N4—C7	−50.7 (4)	P1—N5—C14—C15	−162.9 (4)
O2—P1—N5—C14	37.2 (4)	N5—C14—C15—C16	−177.7 (5)
N6—P1—N5—C14	166.0 (4)	C19—C14—C15—C16	−52.6 (7)
N4—P1—N5—C14	−79.8 (4)	C14—C15—C16—C17	54.8 (8)
O2—P1—N6—C20	−61.0 (4)	C15—C16—C17—C18	−55.0 (8)
N5—P1—N6—C20	171.1 (4)	C16—C17—C18—C19	55.1 (8)
N4—P1—N6—C20	55.2 (4)	N5—C14—C19—C18	175.1 (5)
P1—N4—C7—O3	−7.5 (6)	C15—C14—C19—C18	52.8 (7)
P1—N4—C7—C8	170.5 (3)	C17—C18—C19—C14	−54.7 (8)
O3—C7—C8—C9	147.1 (4)	P1—N6—C20—C21	−124.3 (4)
N4—C7—C8—C9	−31.0 (6)	P1—N6—C20—C25	110.5 (4)
O3—C7—C8—C13	−28.7 (6)	N6—C20—C21—C22	−174.9 (5)
N4—C7—C8—C13	153.3 (4)	C25—C20—C21—C22	−50.1 (7)
C13—C8—C9—C10	1.2 (7)	C20—C21—C22—C23	53.6 (7)
C7—C8—C9—C10	−174.5 (5)	C21—C22—C23—C24	−54.7 (7)
C8—C9—C10—C11	−1.2 (8)	C22—C23—C24—C25	55.0 (7)
C9—C10—C11—C12	0.5 (8)	C23—C24—C25—C20	−52.9 (7)
C10—C11—C12—C13	0.1 (8)	N6—C20—C25—C24	175.3 (5)
C11—C12—C13—C8	−0.1 (8)	C21—C20—C25—C24	49.5 (7)
C9—C8—C13—C12	−0.6 (7)		

Hydrogen-bond geometry (\AA , °)

$D\text{—H}\cdots A$	$D\text{—H}$	$H\cdots A$	$D\cdots A$	$D\text{—H}\cdots A$
N4—H4 ⁱ —O3 ⁱ	0.90 (4)	2.16 (4)	2.988 (4)	154 (3)
N5—H5 ⁱⁱ —O2 ⁱⁱ	0.77 (5)	2.30 (6)	3.019 (5)	156 (6)

Symmetry codes: (i) $x, y+1, z$; (ii) $x, y-1, z$.

supplementary materials

Fig. 1

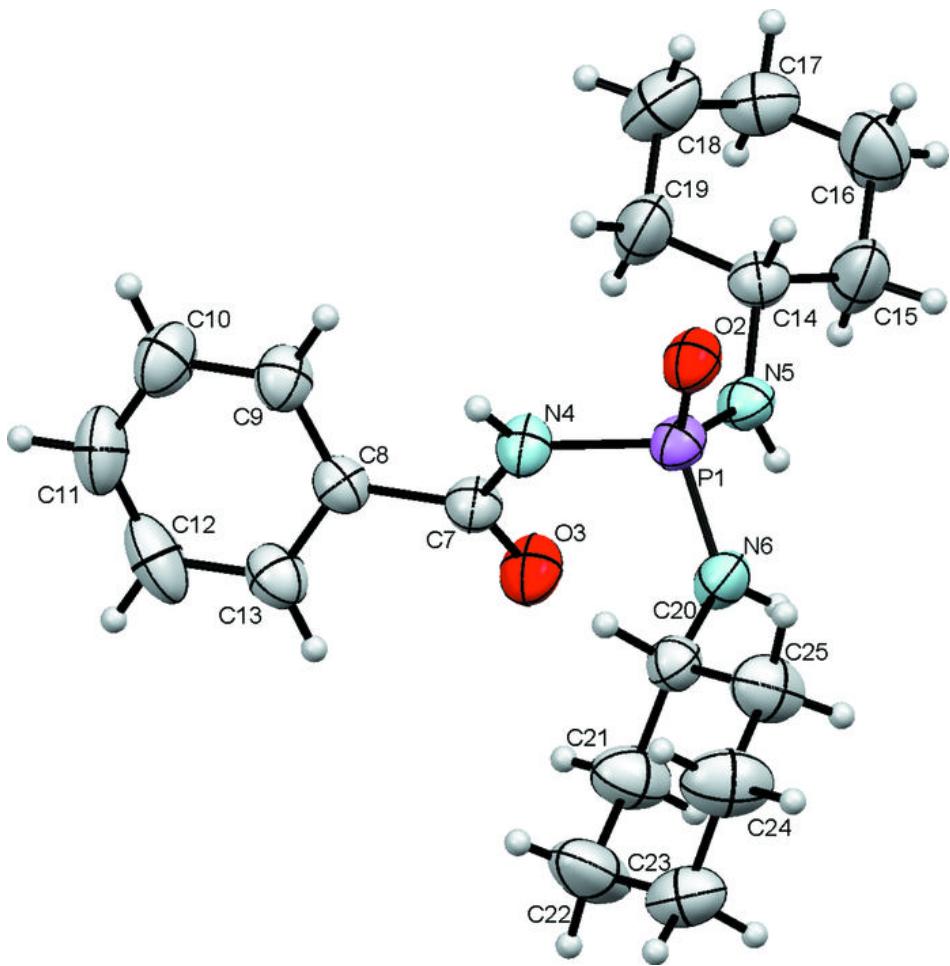


Fig. 2

